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(54) Wood preservative concentrate and an agent prepared therefrom for preserving wood and timber

(57) A water-diluted wood preservative concentrate which does not require a substantial amount of petroleum-derived solvent comprises, by weight, from 5 to 43 parts of at least one organic, water-insoluble insecticide and/or at least one organic fungicide, which may be water-soluble or water-insoluble, from 5 to 35 parts of at least one water-dilutable synthetic resin, from 35 to 5 parts of at least one plasticiser or water-insoluble organic solvent containing hydroxyl and/or ether groups, from 36 to 10 parts of at least one non-ionic emulsifier, from 0 to 6 parts of a buffer compound or pH regulator, and from 0 to 15 parts of water. The concentrate can

be diluted with water in the proportions 10 to 30% of anhydrous concentrate to 90 to 70% water for use in preserving wood or timber.

A process for making the concentrate is described by treating at least the insecticide and/or fungicide, plasticiser and/or solvent and emulsifier at a temperature of 10 to 80°C., and under a pressure of 0.5332 to 1.1332 bars until a solution is formed. The remaining components may be added before or after such treatment.

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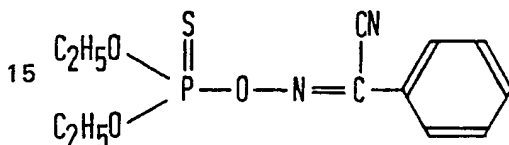
SPECIFICATION

Wood preservative concentrate and an agent prepared therefrom for preserving wood and timber

- 5 This invention relates to wood preservatives and is concerned with a wood preservative concentrate which can be diluted with water to prepare a wood preservative agent for use in preserving wood and timber. The invention also relates to such a wood preservative agent and to a process for preparing the concentrate.
- 10 A wood preservative which is prepared from a liquid concentrate, the active ingredients of which are water-insoluble insecticides and fungicides, which are dissolved in a solvent and in oils, with the addition of emulsifiers, in a manner such that the finished wood preservative is in the form of a mobile emulsion is already known and such a preservative is described in German Offenlegungsschrift No. 2,518,416. The fungicides in this wood preservative are preferably caprylates and naphthenates of heavy metals.
- 15 These wood preservatives have the disadvantage that, when water is added, a milky, turbid emulsion is formed which creams after a certain period of time, and the distribution of the active ingredient is thus not uniform. A sediment could be observed in several cases. In these cases, impregnation of the wood may not be fully effective.
- 20 It is an object of the present invention to provide an agent for preserving wood, in which a considerable percentage of the organic, water-insoluble solvent, which is preferably based on petroleum products, aromatics and the like, can be replaced by other organic substances and/or water, so that a considerable saving can be made in the solvent or the amount thereof. It is desirable that a wood preserving agent should not separate or deposit a sediment during
- 25 processing or storage. In the form of a concentrate the preservative should be stable during storage and transportable, and it should be possible to dilute it to the ready-to-use concentration by simply adding water at the location of use or at the point of distribution. By keeping within certain amounts by weight and matched weight ratios and compositions, the present wood preservative concentrate which can be diluted with water to form a ready-to-use preparation
- 30 should also achieve as adequate as possible a depth of penetration in the wood.
- According to one aspect of the present invention there is provided a water-dilutable wood preservative concentrate, comprising, by weight, the following components:
- a) from 5 to 43 parts of at least one organic water-insoluble insecticide and/or at least one organic fungicide,
 - 35 b) from 5 to 35 parts (calculated as solids content) of at least one water-dilutable synthetic resin,
 - c) from 35 to 5 parts of at least one plasticiser for said resin and/or at least one water-insoluble organic solvent containing hydroxyl and/or ether groups,
 - d) from 36 to 10 parts of at least one non-ionic emulsifier,
 - 40 e) from 0 to 6 parts of a buffer compound or pH regulator, and
 - f) from 0 to 15 parts of water.
- Preferably, the concentrate comprises, by weight,
- a) from 8 to 40 parts of said insecticide and/or fungicide,
 - b) from 15 to 25 parts of said synthetic resin,
 - 45 c) from 25 to 15 parts of said plasticisers and/or solvent,
 - d) from 25 to 15 parts of said emulsifier,
 - e) from 1 to 2 parts of said buffer compound or pH regulator, and
 - f) from 0.001 to 7 parts of water.
- Preferably, the emulsifier component present in the concentrate is a mixture of emulsifiers of which at least one emulsifier is an ethoxylated phenol or an ethoxylated alkyl-, aryl-, or arylalkyl-phenol or an ethoxylated phenol containing one or more other side groups or an ethoxylated organic acid, preferably an ethoxylated nonylphenol or an ethoxylated fatty acid. The emulsifier or mixture of emulsifiers serves to maintain the ingredients of the preservative in a fine distribution and serves to prevent or reduce separation, formation of a sediment or creaming of
- 50 the ingredients. Preferably, the concentrate and the wood preserving agent prepared therefrom contain a mixture of emulsifiers of different chain lengths, of which at least one emulsifier has an ethoxylated side chain with less than 10 ethoxy groups and of which at least one other emulsifier has an ethoxylated side chain with more than 10 ethoxy groups.
- The water-dilutable synthetic resin component is preferably an alkyd resin of medium oil length. The use of such a resin results in better fixing of the active compounds being achieved in spite of the replacement of a substantial amount of the organic solvent based on petroleum, and depending upon the synthetic resin content, makes film formation possible.
- The buffer compound or pH regulator for adjusting the pH value is preferably an amine, an ammonium compound, a quaternary ammonium compound or a mixture of two or more thereof.
- 65 The insecticide component, which may be present in the wood preservative may be a single

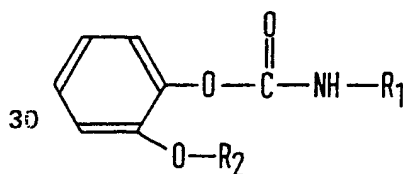
insecticid or a mixture of insecticides and is preferably a carbamate, a phosphoric acid ester, a phosphonic acid ester, a thiophosphoric acid ester, a dithi phosphoric acid ester or a thionophosphoric acid ester, a chlorinated hydr carbon, a pyrethroid and/or endosulphan or a mixture of two or more of these compounds.

- 5 Dieldrin, aldrin and lindane are preferably employed as the chlorinated hydrocarbons, while the preferred pyrethroids include allethrin, cyclethrin, furethrin, tetramethrin, resmethrin, permethrin, decamethrin and bioresmethrin and other insecticidal active compounds of pyrethrum. The preferred insecticidal carbanates include o-isopropoxyphenyl N-methyl-carbamate and/or o-sec.-butylphenyl N-methyl-carbamate.
- 10 Insecticidal thionophosphoric acid esters which may be used include insecticidal thionophosphoric acid esters of the formula

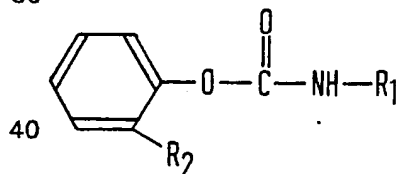


- 20 which may be halogenated, preferably (diethoxy-thiophosphoryloxyimino)-phenylacetonitrile or O,O-diethyl O-(α-cyanobenzylidene-amino)-thionophosphate and (diethoxythiophosphoryloxyimino)-2-chlorophenylacetonitrile, and other phosphoric acid esters, such as dimethoxy-O-(4-nitrom-tolyl)-phosphorothioate.

Carbamate insecticides which may be used and which are soluble in oily or oil-like solvents include alkoxyphenyl N-alkylcarbamates of the general formula

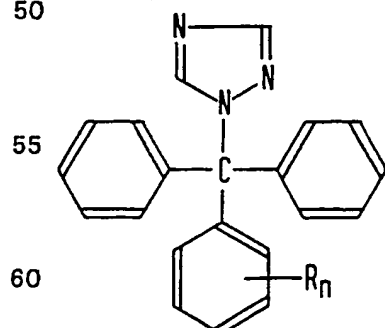


and alkylphenyl N-alkylcarbamates of the general formula



- where, in each case, R₁ is an alkyl radical with 1 to 4 carbon atoms, preferably a methyl radical, and R₂ is an alkyl radical with 1 to 5 carbon atoms, preferably an alkyl radical with 3 or 4 carbon atoms.

The fungicide component which may be present in the wood preservative may be water-insoluble and may be a single fungicide or a mixture of said fungicides. The fungicide is preferably a tetravalent organotin compound, a chlorinated phenol, preferably penta- or tetrachlorophenol, a 1-trityl-1,2,4-triazole of the general formula



- in which R is a fluorine, a chlorine or bromine atom, a trifluoromethyl, nitro or cyano group or an alkyl group with up to 4 carbon atoms and n is 1 or 2, or a salt of such a triazole with an organic or inorganic acid, or an (N-cyclohexyl-diazoniumdioxy)-metal compound (i.e. a salt of N-

nitroso-N-cyclohexylhydroxylamine), preferably the aluminium compound thereof, or N,N-dimethyl-N'-phenyl-N'-(fluorodichloromethylthio)-sulphamide or N,N-dimethyl-N'-p-tolyl-N'-(dichlorofluoromethylthio)-sulphamide, or a mixture of two or more of these compounds.

Examples of oil-soluble tetravalent, fungicidal organotin compounds which can be employed include tributyltin benzoate, tris-(tributyl-tin) phosphate and bis-(tributyl-tin) oxide.

Alternatively, the fungicide component of the present composition may be water-soluble and, in this case, the preferred water-soluble fungicides are chlorophenolates, and water-soluble metal salts of an N-cyclohexyl-diazoniumdioxo compound (or a salt of N-nitroso-N-cyclohexylhydroxylamine). These fungicides may also be used singly or as a mixture of two or more thereof.

The water-insoluble organic solvents containing hydroxyl groups and/or ether groups which may be used in the present compositions include water-insoluble polyols or ethers or esters of polyols, preferably glycol ethers.

A plasticiser or mixture of plasticisers which is liquid at room temperature may be used as the plasticiser ingredient of the present composition. The plasticiser is preferably an alkyl, aryl or aryl alkyl phthalate, preferably dibutyl phthalate, dioctyl phthalate or benzyl butyl phthalate; an alkyl phosphate or phosphoric acid ester, preferably tributyl phosphate; or an adipate, preferably di-(2-ethylhexyl) adipate; a stearate and/or oleate, for example an alkyl stearate or alkyl oleate, preferably butyl oleate, butyl stearate or amyl stearate; or a liquid glycerol ester. The plasticisers may be present singly or as a mixture of two or more thereof in the composition.

For certain recipes, it may be desirable to add an antifoaming agent, such as a silicone antifoaming agent or an alkyl phosphate, preferably tri-n-butyl phosphate to the composition.

The ready-to-use wood preserving agent, prepared from the wood preservative concentrate by diluting the latter with water, consists of 10 to 30% by weight, preferably 15 to 25% by weight, of the concentrate (calculated as anhydrous concentrate) and 90 to 70% by weight, preferably 85 to 75% by weight, of water.

The wood preservative can be applied to the wood by methods which are known per se, such as by brushing, spraying, atomising or using impregnating methods, for example, immersion, pressure and/or vacuum methods.

The invention furthermore relates to a process for the preparation of the wood preservative concentrate and of the agents prepared therefrom for preserving wood and timber products. Accordingly, another aspect of the present invention provides a process for the preparation of the wood preservative concentrate wherein the insecticide and/or fungicide component which is soluble in the plasticiser or solvent component and the non-ionic emulsifier component are treated at a temperature of 10 to 80°C. and under a pressure of 400 mm. Hg to 850 mm. Hg (0.5332 to 1.1332 bars) in the presence or absence of the remaining components until a solution is formed, whereafter the one or any remaining components, including the buffer compound and, if present, water, are added to the solution. Preferably the treatment is effected at a temperature of 30 to 60°C., and under a pressure of 600 mm. Hg to 790 mm. Hg (0.7999 to 1.0532 bars).

The invention will now be illustrated by the following Examples of wood preservative concentrates which can be diluted with water and have an insecticidal and fungicidal action.

Example 1

45	Lindane)	2% by weight	45
	Aldrin) (insecticide)	1% by weight	
	Sodium pentachlorophenolate	(fungicide)	35% by weight	
50	Alkyd resin which can be diluted with water		14% by weight	50
	Butylglycol (solvent)		6% by weight	
	Dibutyl phthalate (plasticiser)		20% by weight	
	Ethoxylated nonylphenol: (emulsifier)			
55	Degree of ethoxylation	6	7% by weight	55
		10	7% by weight	
		14	7% by weight	
	Amine (pH regulant)		1% by weight	
60	Use: 20% of concentrate to 80% of water.			60

A ready to-use wood preservative was obtained by mixing 20% by weight of the concentrate with 80% by weight of water.

Example II

5	o,o-Diethyl o-(α -cyanobenzylideneamino)-thionophosphate (phoxim)	8% by weight	5
	Sodium pentachlorophenolate	25% by weight	
	Alkyd resin which can be diluted with water	17.5% by weight	
	Butylglycol	7.5% by weight	
10	Dibutyl phthalate	20.0% by weight	10
	Ethoxylated nonylphenol:		
	Degree of ethoxylation 6	7.0% by weight	
	10	7.0% by weight	
	12	7.0% by weight	
15	Amine	1.0% by weight	15
	Use: 20% of concentrate to 80% of water.		

A ready-to-use wood preservative was obtained by mixing 20% by weight of the concentrate with 80% by weight of water.

20 *Example III* 20

	Lindane	4.0% by weight	
	(N-Cyclohexyl-diazoniumdioxy)-potassium	5.0% by weight	
25	Alkyd resin which can be diluted with water	14.0% by weight	25
	Butylglycol	6.0% by weight	
	Ethylglycol	22.5% by weight	
	Ethylglycol acetate	32.5% by weight	
30	Ethoxylated nonylphenols:		30
	Degree of ethoxylation 6	5.0% by weight	
	10	5.0% by weight	
	14	5.0% by weight	
	Amine	1.0% by weight	
35	Use: 15% of concentrate to 85% of water.		35

A ready-to-use wood preservative was obtained by mixing 15% by weight of the concentrate with 85% by weight of water.

40 CLAIMS 40

1. A water-dilutable wood preservative concentrate, comprising, by weight, the following components:
 - a) from 5 to 43 parts of at least one organic, water-insoluble insecticide and/or at least one organic fungicide,
 - 45 b) from 5 to 35 parts (calculated as solids content) of at least one water-dilutable synthetic resin,
 - c) from 35 to 5 parts of at least one plasticiser for said resin and/or at least one water-insoluble organic solvent containing hydroxyl and/or ether groups,
 - d) from 36 to 10 parts of at least one non-ionic emulsifier,
 - 50 e) from 0 to 6 parts of a buffer compound or pH regulator, and
 - f) from 0 to 15 parts of water.
2. A wood preservative concentrate as claimed in claim 1, comprising, by weight,
 - a) from 8 to 40 parts of said insecticide and/or fungicide,
 - b) from 15 to 25 parts of said synthetic resin,
 - 55 c) from 25 to 15 parts of said plasticiser and/or solvent,
 - d) from 25 to 15 parts of said emulsifier,
 - e) from 1 to 2 parts of said buffer compound or pH regulator, and
 - f) from 0.001 to 7 parts of water.
3. A wood preservative concentrate as claimed in claim 1 or 2, wherein the emulsifier is a mixture of emulsifiers of which at least one is an ethoxylated phenol, an ethoxylated alkyl-, aryl-, 60 or arylalkyl-phenol, an ethoxylated phenol containing one or more other side groups or an ethoxylated organic acid.
4. A wood preservative concentrate as claimed in claim 3, wherein the emulsifier is an ethoxylated nonylphenol or an ethoxylated fatty acid.
- 65 5. A wood preservative concentrate as claimed in claim 1 or 2, wherein the emulsifier is a 65

mixture of emulsifiers of which at least one has an ethoxylated side chain with less than 10 ethoxy groups and of which at least one other has an ethoxylated side chain with more than 10 ethoxy groups.

6. A wood preservative concentrate as claimed in any one of claims 1 to 5, wherein the synthetic resin is an alkyd resin of medium oil length. 5

7. A wood preservative concentrate as claimed in any one of claims 1 to 6, wherein the buffer material or pH regulator is an amine, an ammonium compound, a quaternary ammonium compound, or a mixture of two or more thereof.

8. A wood preservative concentrate as claimed in any one of claims 1 to 7, wherein the insecticide is a carbamate, a phosphoric acid ester, a phosphonic acid ester, a thiophosphoric acid ester, a dithiophosphoric acid ester, a thionophosphoric acid ester, a chlorinated hydrocarbon, a pyrethroid or endosulphan, or a mixture of two or more of these compounds. 10

9. A wood preservative concentrate as claimed in any one of claims 1 to 8, wherein the fungicide is water-insoluble.

10. A wood preservative concentrate as claimed in claim 9, wherein the water-insoluble fungicide is a tetravalent organotin compound, a chlorinated phenol, a 1-phenyl-1,2,4-triazole of the general formula 15



in which R is a fluorine, chlorine or bromine atom, a trifluoromethyl, nitro or cyano group or an alkyl group with up to 4 carbon atoms and n is 1 or 2, or a salt of such a triazole with an organic or inorganic acid, or a (N-cyclohexyl-diazoniumdioxy)-metal compound (or a salt of N-nitroso-N-cyclohexylhydroxylamine), N,N-dimethyl-N'-phenyl-N'-(fluorodichloromethylthio)-sulphamide, N,N-dimethyl-N'-p-tolyl-N'-(dichlorofluoromethylthio)-sulphamide, or a mixture of two or more of these compounds. 20 25 30

11. A wood preservative concentrate as claimed in claim 10, wherein the chlorinated phenol is pentachlorophenol or tetrachlorophenol.

12. A wood preservative concentrate as claimed in claim 10, wherein said (N-cyclohexyl-diazoniumdioxy)-metal compound is the aluminium compound. 40

13. A wood preservative concentrate as claimed in any one of claims 1 to 8, wherein the fungicide is water-soluble.

14. A wood preservative concentrate as claimed in claim 13, wherein the water-soluble fungicide is a water-soluble phenolate, and/or water-soluble metal salt of a N-cyclohexyl-diazoniumdioxy compound (or a salt of N-nitroso-N-cyclohexylhydroxylamine). 45

15. A wood preservative concentrate as claimed in claim 14, wherein the phenolate is a chlorophenolate.

16. A wood preservative concentrate as claimed in claim 15, wherein the water-insoluble organic solvent containing hydroxyl and/or ether groups is a water-insoluble polyol or an ether or ester of a polyol. 50

17. A wood preservative concentrate as claimed in claim 16, wherein said solvent is a glycol ether.

18. A wood preservative concentrate as claimed in any one of claims 1 to 17, wherein the plasticiser is a plasticiser or mixture of plasticisers which is liquid at room temperature.

19. A wood preservative concentrate as claimed in any one of claims 1 to 18 wherein the plasticiser is an alkyl, aryl or aryl alkyl phthalate, an alkyl phosphate or phosphoric acid ester, or an adipate, a stearate or oleate, a liquid glycol ester or a mixture of two or more thereof. 55

20. A wood preservative concentrate as claimed in claim 19, wherein the phthalate plasticiser is dibutyl phthalate, dioctyl phthalate or benzyl butyl phthalate, wherein the phosphate plasticiser is tributyl phosphate, wherein the adipate plasticiser is di-(2-ethylhexyl)adipate, or wherein the stearate or oleate plasticiser is butyl oleate, butyl stearate or amyl stearate. 60

21. A wood preservative concentrate substantially as hereinbefore described in any one of the foregoing Examples.

22. A wood preservative agent for use in preserving wood or timber, comprising from 10 to 30% by weight of the concentrate claimed in any preceding claim, calculated as anhydrous 65

concentrat , and from 90 to 70% by weight of water as diluent.

23. A wood preservative agent as claimed in claim 22, comprising from 15 to 25% by weight of the concentrate and from 85 to 75% by weight of water.

24. A process for the preparation of the wood preservative concentrate claimed in any one of claims 1 to 21, wherein the insecticide and/or fungicide component which is soluble in the plasticiser or solvent component and the non-ionic emulsifier component are treated at a temperature of 10 to 80°C., and under a pressure of 400 mm. Hg to 850 mm. Hg (0.5332 to 1.1332 bars) in the presence or absence of the remaining components until a solution is formed, whereafter the one or any remaining components, including the buffer compound and, if present, water, are added to the solution.

25. A process as claimed in claim 24, wherein the treatment is effected at a temperature of 30 to 60°C., and under a pressure of 600 mm. Hg to 790 mm. Hg (0.7999 to 1.0532 bars).

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